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DEVELOPMENT OF MODEL-BASED MAGNETIC LP-LEC GROWTH OF LARGE DIAMETER GAAS

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The objective of this research was to explore the range of analytical information on the defect structure of doped and semi-insulating GaAs obtainable from computational, non-invasive, near infrared absorption analysis. Motivation for this research was provided by the realization that the establishment of meaningful property specifications for device materials is contingent on non-invasive defect analysis executable in a fabline environment. Infrared absorption measurements on a micro- and macro-scale in combination with computational image processing and analysis were found to meet the requirements of the stated research objectives.

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DEVELOPMENT OF MODEL BASED MAGNETIC LP–LEC GROWTH OF LARGE DIAMETER GaAs

Final Technical Report

AFOSR-91-0355

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CONTENTS

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INTRODUCTION

The objective of this research was to explore the range of analytical information on the defect structure of doped and semi-insulating GaAs obtainable from computational, non-invasive, near infrared absorption analysis. Motivation for this research was provided by the realization that the establishment of meaningful property specifications for device material is contingent on non-invasive defect analysis executable in a fabline environment. Infrared absorption measurements on a micro- and macro-scale in combination with computational image processing and analysis were found to meet the requirements of the stated research objectives.

SUMMARY

Optical semiconductor characterization by computational NIR absorption analysis has been found to be an effective approach to the determination of the following properties:

- dopant concentration and distribution (macroscale)
- local segregation effects (microscale)
- residual macro-stress
- dislocation density and distribution
- stress, associated with dislocations
- precipitates
- annealing effects
- subsurface damage

The broad spectrum of defect identification was achieved by complementing conventional bright field transmission microscopy with dark field, phase contrast and polarized light microscopy.

The approach taken is characterized by its non-invasive nature, applicability on a macro- and micro-scale, high resolution, rapid execution and digital data storage with spatial coordinates suitable to conduct subsequent correlation analyses with spatial distribution of device deficiencies.

APPROACH TO RESEARCH

The analytical technique developed for rapid, non-destructive defect analysis in semiconductors is based on quantitative image analysis in conjunction with near infrared (NIR) transmission microscopy. NIR microscopy provides an image of the semiconductor sample (0.5-2 mm thick) reflecting any local variations in the absorption coefficient across the field due to the presence of defects. Qualitatively this image provides an excellent representation of material uniformity. It contains in addition, however, information which can be used for the quantitative determination of local free charge carrier concentrations and lattice stress conditions, for example.

NIR microscopy relies on an imaging device such as a CCD camera or a silicon vidicon camera to detect transmitted radiation. To quantify the image, the output of the camera is used as input to a digital image processor where the signal is digitized into a 512 by 480 pixel array with a dynamic range of 256 gray levels. The image processing system provides for multiple image storage and near real time whole image mathematics capability. The approach taken for data calibration is to increase the gain of the system so that the entire range is distributed over the limited transmittance range exhibited by the sample; using neutral density filters, the measured gray level value can then be equated to transmittance. Comprehensive defect mapping can be accomplished by determination of the IR (and, as applicable, the visible) spectrum for a given material by means of an FTIR spectrometer and identification of chemical defects, for example, through their characteristic absorption peaks (fig. 1). The spatial distribution of these defects is subsequently analyzed and the corresponding data are stored in digitized form with their spatial coordinates. The fundamental characteristics of computational optical defect analysis are presented in fig. 2. The mode is non-invasive, applicable to both the micro- and macro-scale with a maximum spatial resolution approaching 1 μm; data storage with spatial coordinates is provided for with complete image analysis requiring a fraction of one second.

Computational image analysis has been applied to dopant concentration analysis, stress analysis, dislocation density measurements in conducting and semi-insulating matrices, surface damage measurements and precipitation analyses. Work is in progress on the determination of lattice damage associated with ion implantation and on the identification of defect propagation into epitaxial layers.

The non-destructive nature of the computational image analysis in combination with digital data storage provides a means for the conduct of a statistical correlation analysis between device and spatially coincident wafer characteristics. Such analyses can be accomplished upon subjecting the analyzed wafer to device processing and mapping the intermediate processing steps as well as the spatial distribution of yield and performance of devices (fig. 3).

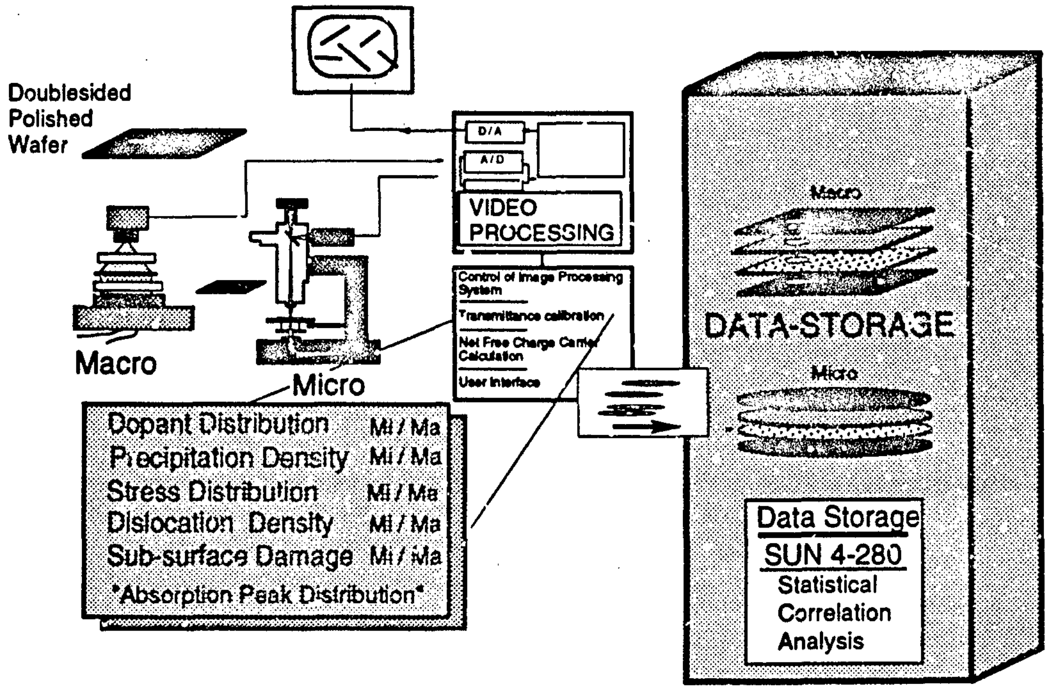
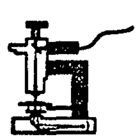


Fig.1 Computational NIR Absorption Analysis with Data Storage

Characteristics:

* Mode: Non-Invasive
* Scale: Macro, Selected Micro
* Resolution: (as applicable) Micron to Sub-Micron Range
* Data Storage: Digital with Spatial Coordinates
* Analysis Time: Fractional Seconds

Properties revealed by non-invasive Optical analysis:



Dopant Concentration and Distribution (macro)
Local Segregation Inhomogeneities (micro)
Residual Stress Distribution (macro)
Dislocations (micro)
Stress Associated with Dislocations (micro)
Precipitates (micro)
Surface Damage (macro, micro)
Annealing Effects (macro, micro)
Absorption peaks in visible and NIR range

Fig.2 Capabilties of Semiconductor Characterization by Computational Absorption Analysis

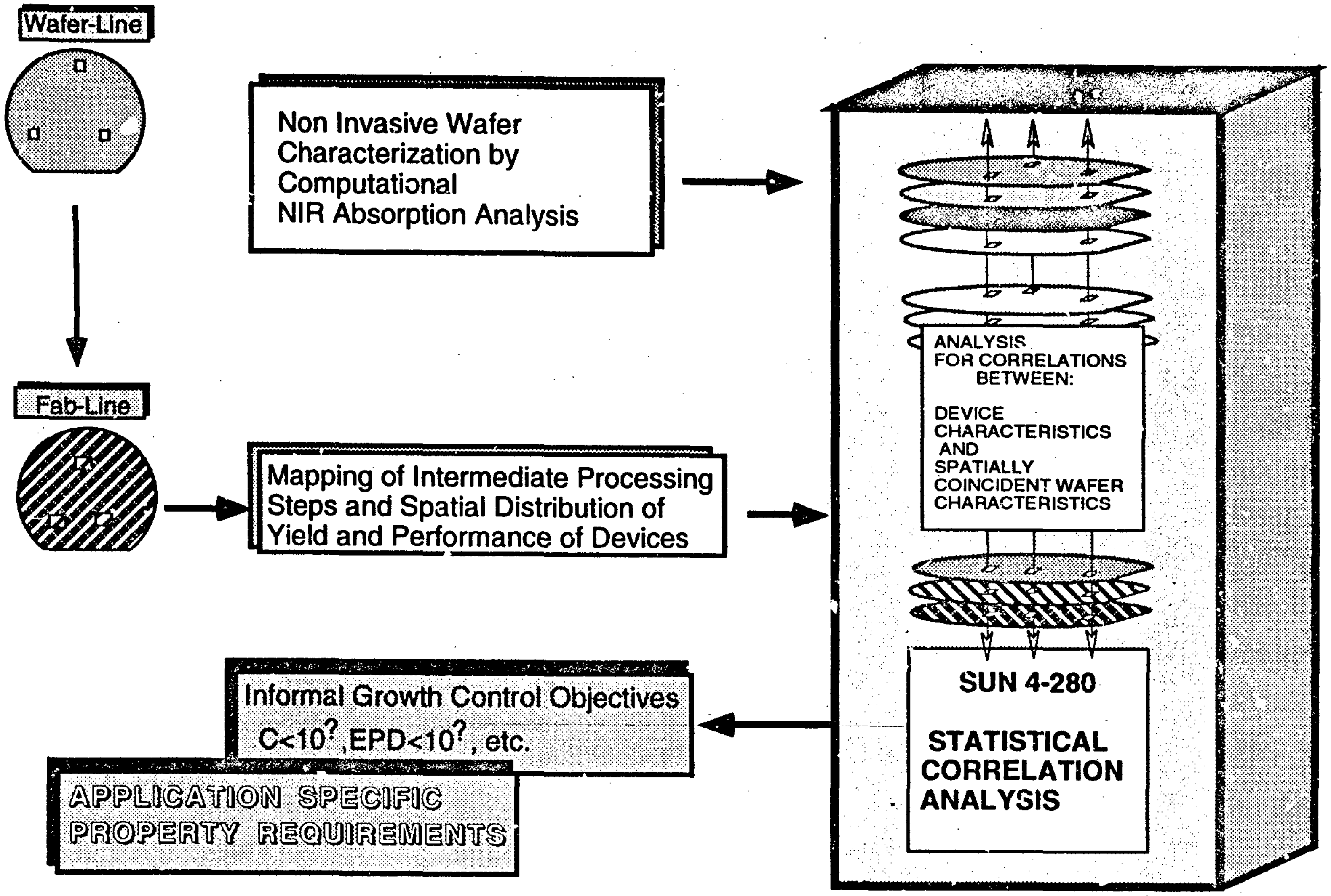


Fig. 3 Establishment Of User Specified Property Requirements Through Statistical Correlation Analysis

RESULTS

An NIR transmission micrograph of commercial Te-doped GaAs with a corresponding free charge carrier (dopant) distribution analysis is given in fig. 4. (The section analyzed was cut from a crystal along its growth axis.) The quantitative microscale analysis reveals that microsegregation effects associated with LP-LEC growth of GaAs result in compositional fluctuations which, over micron dimensions, approach and in some instances exceed one order of magnitude. This finding is contrasted by the general belief that such fluctuations are in the range of ±15 to 30% of the average doping value. It is also found that, contrary to expectation, the striation pattern is discontinuous along the crystal diameter. On the basis of this quantitative segregation analysis, it is concluded that growth is subjected to turbulent free density driven melt convection; this results in localized thermal perturbations which penetrate the solute boundary layer and lead to melt-back. Subsequent regrowth and dopant incorporation appear largely controlled by fluid dynamics at the phase boundary and cannot be interpreted on the basis of the Burton, Prim and Slichter theory. A transmission micrograph of a GaAs segment cut normal to the growth axis (conventional wafer geometry) and polished on both sides was subjected to dislocation analysis (fig. 5). The visibility of the complex dislocation network is attributed to decoration of the dislocations by dopant elements. It should be pointed out that a comparative analysis of dislocations in GaAs grown by the LEC technique with GaAs grown by the horizontal Bridgman technique revealed fundamental differences in density, distribution and geometry (fig. 6).

Analysis of semi-insulating GaAs indicated transparency, absence of contrast generating media, to 1 μm radiation in bright field transmission mode. Using dark field NIR illumination, extensive scattering centers of submicron dimensions are observed. The scattering centers, precipitates, appear as decorations of dislocations and thus delineate the location of these otherwise invisible defects (fig. 7).

Unambiguous identification of dislocations in semiconductors has thus far been accomplished primarily by electron microscopy and X-ray topography. These approaches are destructive and/or time consuming respectively, thus finding limited application. The third and most widely used approach to dislocation identification, chemical etching, although destructive in nature, is convenient because of its simplicity. Its use is based on the fundamental assumption that the termination of dislocations on the surface gives rise to a local modification of the etch rate and thus results in the generation of etch pits. Etch pit densities are equated with dislocation densities.

There exits in most instances uncertainty as to the one-to-one correspondence between dislocations and etch–pits; chemical etchants can also generate pits that are not related to dislocations and, on the other hand, there is no assurance that all dislocations are revealed through etch pits.

Using near IR bright field transmission microscopy, dislocations in n-type GaAs grown by the horizontal Bridgman technique are revealed in a non-destructive manner through contrast due to impurity decoration of the dislocation lines (fig. 6). By subjecting the same wafer to photo-etching, etch pits are generated which can be identified (and counted) using interference contrast microscopy with white light in reflection mode (fig. 8a). Operating the microscope in transmission mode, the same sample area yields the transmission image shown in fig. 8b. Accordingly it is established that each etch pit on the surface is associated with a dislocation and, on the other hand, that the termination of each (decorated) dislocation is an etch pit.

It should be pointed out that the establishment of correspondence between dislocations and etch pits cannot be generalized. It must be verified for each particular material system and for the specific etching procedure used.

In the last phase of this research effort commercial GaAs wafers were analyzed for surface damage. It was found that all wafers fail to exhibit processing induced surface and subsurface damage when viewed in reflected NIR Nomarski interference contrast. All wafers, on the other hand, do exhibit varying degrees of subsurface damage when viewed in phase contrast NIR transmission mode (fig. 9).

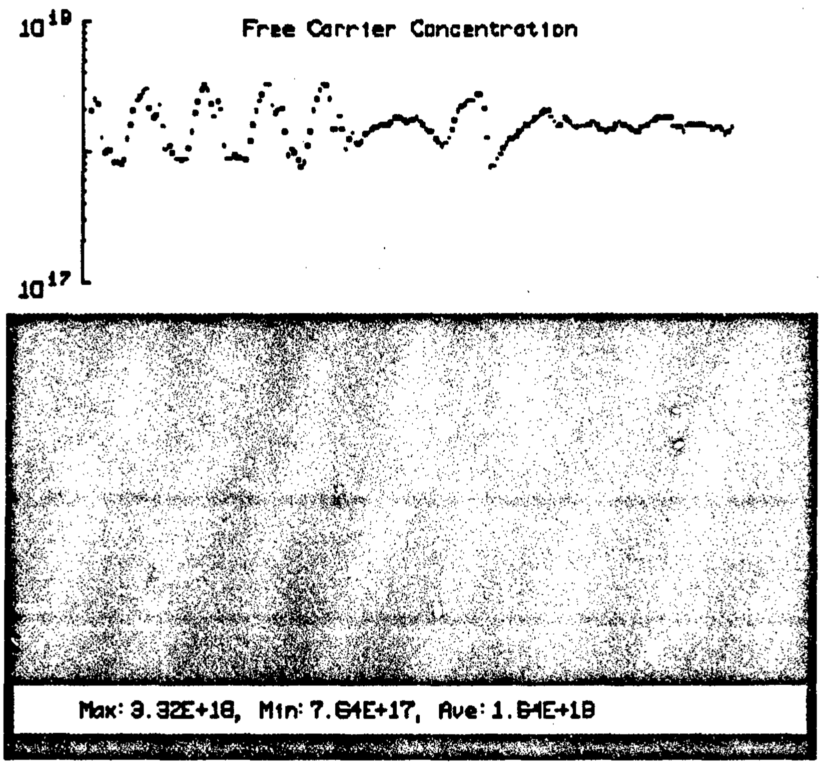


Fig.4 Quantitative Dopant Segregation Analysis in M-LEC GaAs Based on NIR Absorption

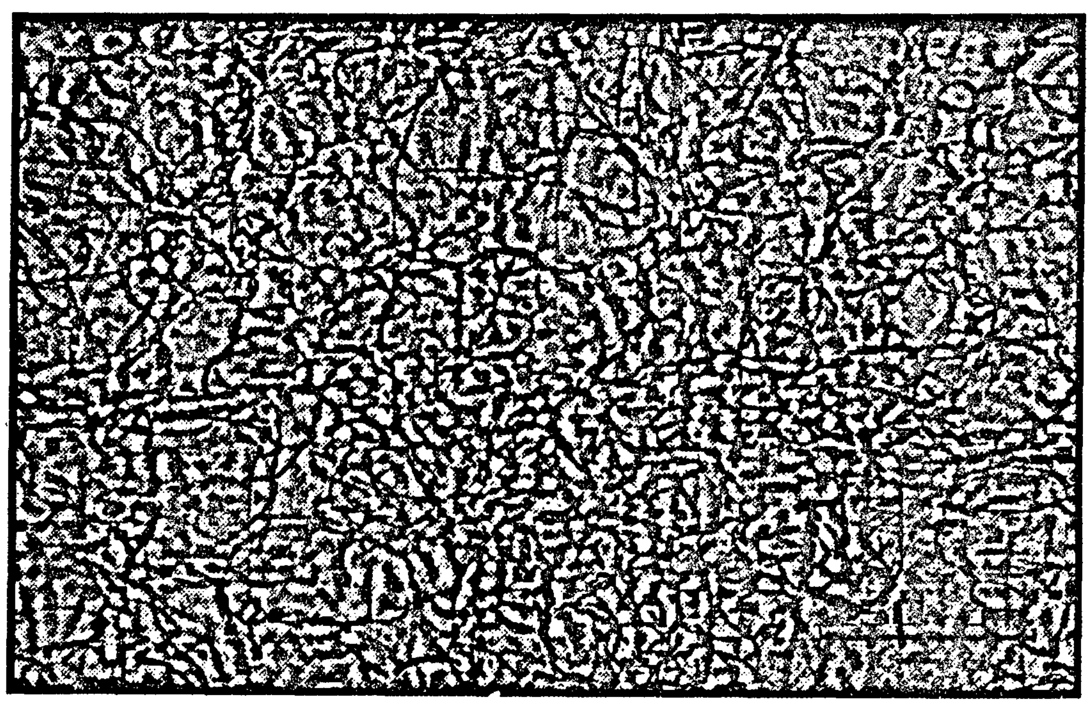


Fig.5 Dislocation Network in Doped LEC GaAs as Revealed by NIR Transmission Microscopy

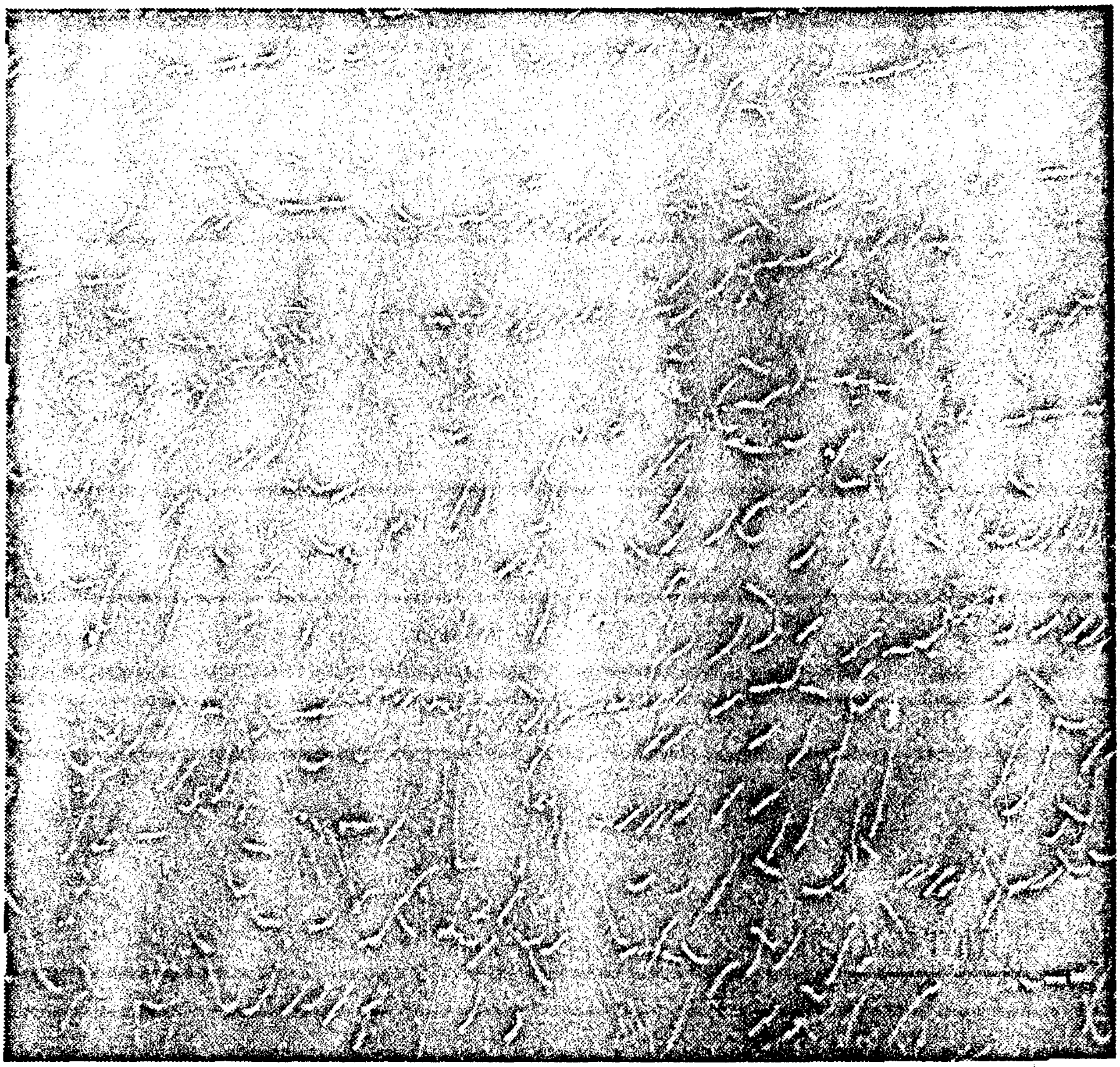


Fig.6 Dislocations in n-type GaAs grown by the horizontal Bridgman technique, as revealed by NIR transmission microscopy. (Compare with dislocation network in LEC GaAs.)

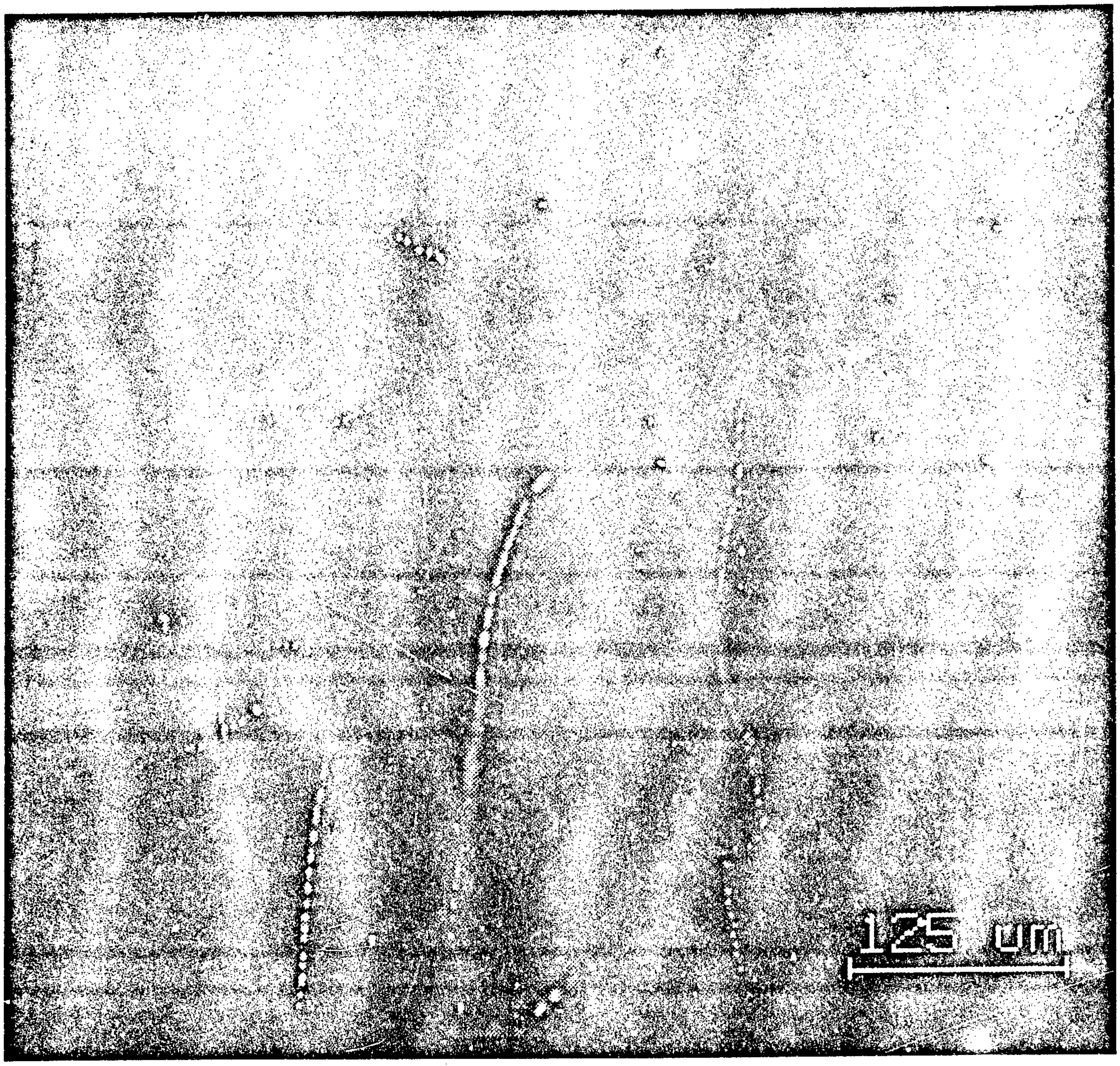


Fig.7 Dislocations as Revealed through Decorating Precipitates in NIR Transmission Darkfield Microscopy.

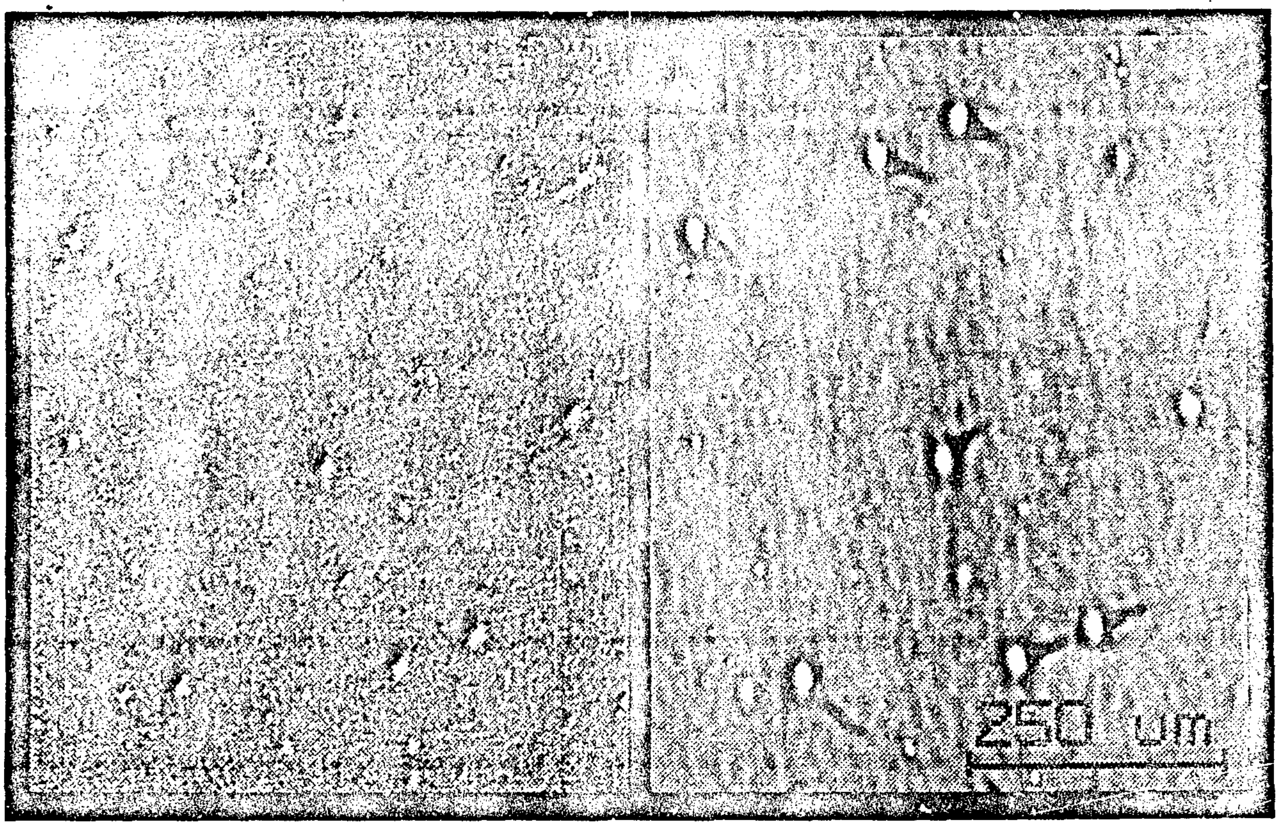


Fig.8 (a) GaAs grown by the horizontal Bridgman technique; the wafer surface shown was subjected to photo-etching; notice etch pits, presumed to correspond to surface terminations of dislocations.
(b) The same area of the wafer as shown in (a) observed in NIR bright field transmission mode; notice the dislocation lines extending from each etch pit.

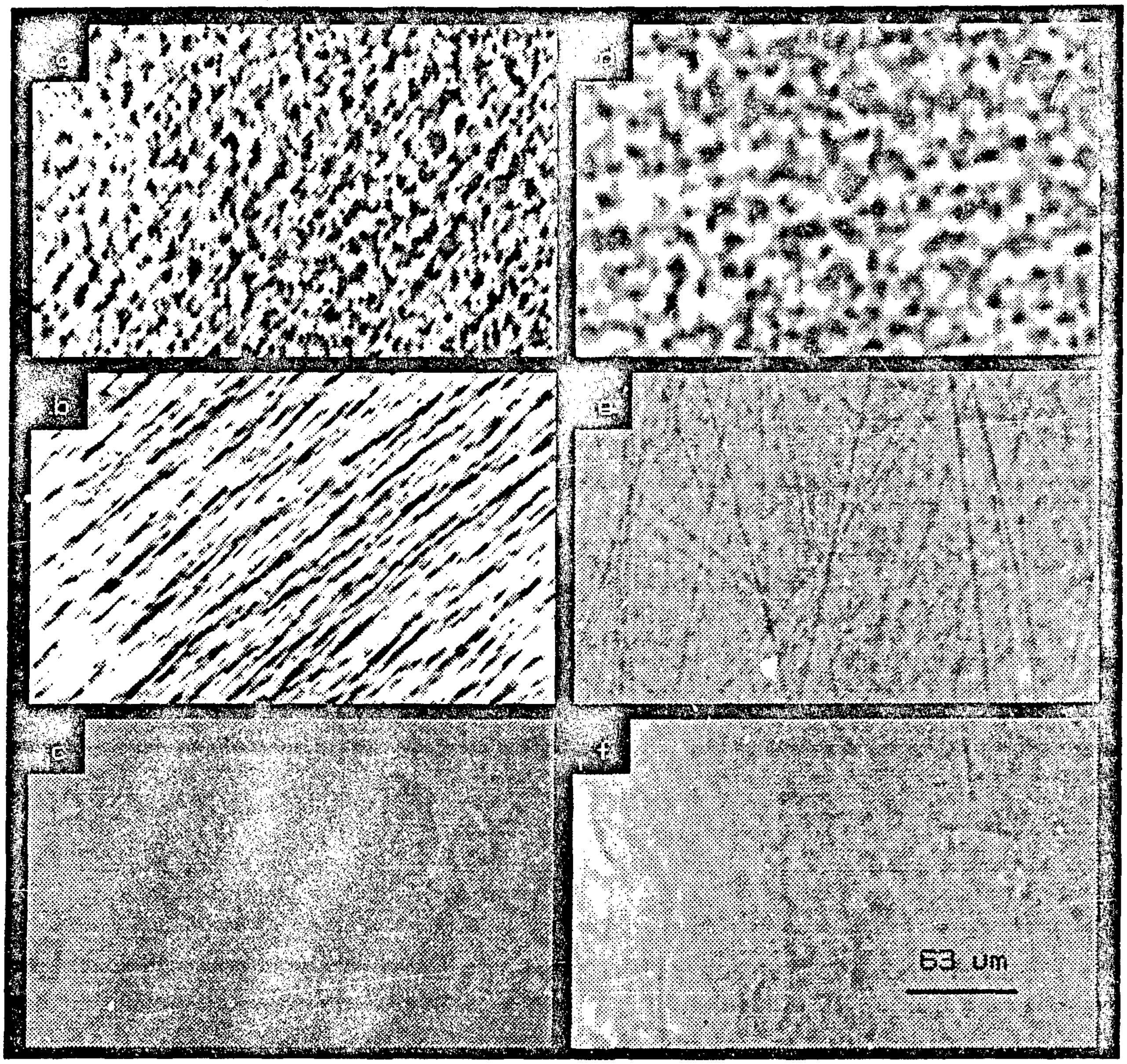


Fig.9 Sub-Surface Damage in Commercial GaAs Wafers as Revealed by NIR Phase Contrast Microscopy in Transmission Mode. (None of the wafers revealed any surface deficiencies when viewed in reflected light Nomarski-type interference contrast microscopy)

CONCLUSIONS

NIR transmission microscopy combined with computational image analysis provides a means for quantitative defect analysis with high sensitivity and spatial resolution. The developed procedures provide micro- and macro-scale analyses which can contribute to an elucidation of growth and segregation phenomena. The sensitivity and resolution achieved permits the identification of gravitational effects on crystal perfection resulting during growth from the melt and from solution. Compatibility of the analytical procedure with tele-operation has been established and is expected to provide for a major advance in efforts to explore the potential of reduced gravity environment for electronic materials research.

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2. X.Z. Cao and A.F. Witt, "Decorated Dislocations in SI-GaAs as Revealed by Dark Field NIR Transmission Microscopy", J. Crystal Growth 112, 838-840 (1991).

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5. M.J. Wargo and A.F. Witt, "Real Time Thermal Imaging for Analysis and Control of Crystal Growth by the Czochralski Technique", J. Crystal Growth 116 (1/2) 213-224 (1992).

PRESENTATIONS

1. "Emerging Issues in Semiconductors", IAP Course 3.093, MIT, January 24, 1992.

2. "What's New in Semiconductor Growth and Characterization", MESS Seminar, MIT, February 27, 1992.

3. "Non-Invasive Bulk Characterization of GaAs and Si Wafers", Hewlett-Packard, Inc., San Jose, CA, March 4, 1992.

4. "Characterization of GaAs Wafers", Intel Corporation, Santa Clara, CA, March 4, 1992.

5. "Non-Invasive Wafer Characterization", Motorola Corporation, Tempe, AZ, March 6, 1992.

6. "New Approaches to Non-Invasive Semiconductor Characterization", Tenth International Conference on Crystal Growth, San Diego, CA, August 18, 1992.

7. "An Analysis of Status, Problems and Opportunities in Crystal Growth At-large and Controlled Solidification in Particular", EuroCryst Meeting, Vienna, Austria, November 27, 1992.

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